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CEMENT LABORATORY MANUAL

A MANUAL OF INSTRUCTIONS FOR THE USE OF STUDENTS IN CEMENT LABORATORY PRACTICE

BY

L. A. WATERBURY, C.E.

PROFESSOR OF CIVIL ENGINEERING, UNIVERSITY OF ARIZONA

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PREFACE

THIS manual has been prepared for the use of students taking the course in cement laboratory practice in the University of Illinois, and for the use of others who may have occasion to use such a laboratory manual. Instructions for the problems originally used in the course mentioned were devised by Ira O. Baker, Professor of Civil Engineering, University of Illinois, under whose direction the author had charge of the cement laboratory at that institution for three years. This manual has been prepared by revising and extending the instructions already in use. The problems which are given herein are suitable to class use and are not intended to serve as instructions for the testing of cements for commercial purposes. However, the problems have been designed to include all of the tests which are ordinarily made, so that a student who shall have completed these problems should be able to do testing for commercial purposes, although the experience which is required for the production of uniformly satisfactory results in the latter class of work can be obtained only by a considerable amount of practice, and cannot be obtained to any considerable extent by a laboratory

course which is intended chiefly to teach methods of testing.

The writer is very much indebted to Professor Ira O. Baker for suggestions and assistance in the preparation of this manual.

L. A. W.

July, 1908.

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CEMENT LABORATORY MANUAL

CHAPTER I.

GENERAL INSTRUCTIONS.

ART. 1. LABORATORY WORK.

1. **Methods.** There is probably no similar class of work in which there is greater necessity for carefulness than in the testing of cement. The results obtained by two observers who follow the same instructions may vary considerably, and the results which may be obtained by different methods of manipulation are likely to differ by more than 100 per cent. Therefore, to obtain results which correctly indicate the character of the cements which are tested, it is essential that there shall be uniformity, not only in the general methods used, but also in the details of the tests. The specifications which have been recommended by various organizations, such as the United States Army Engineers, the Institution of Civil Engineers, the Canadian Society of Civil Engineers, and others, have prescribed methods which should be used in making some of the necessary tests. However, none of these specifications include a complete set of directions

for making all of the required tests. The rules of the American Society of Civil Engineers proposed in 1885 were also deficient. In order to obtain a more complete and better set of rules the American Society of Civil Engineers in 1897 appointed a committee on Uniform Tests of Cement. The methods recommended by the committee were embodied in a progress report which was presented at the annual meeting of the Society, January 21, 1903, and which was amended at the annual meeting, January 20, 1904, and at the annual meeting January 15, 1908. The methods which were recommended are given in Appendix I of this manual. The methods prescribed in the problems herein contained include these standard methods, although some other methods are also described, both for the purpose of comparison and also for the purpose of familiarizing the student with methods which are in use or which have been in use by various authorities.

2. Since the student will not be able to devote a great amount of time to each test, it is necessary that he should note carefully the prescribed methods. He should observe whether the methods are in use in this country or elsewhere, and by whom they are recommended. He should observe the differences in methods of operation, and should try to determine the effect of such differences upon the results. The student should also notice the various kinds of apparatus in use, and should try to determine

which produce the most reliable results and which are the most economical in time required for operation.

3. **Care of Apparatus.** In using the apparatus considerable care is required to avoid breaking the glassware. Flasks and beakers are usually made of very thin glass, and are very likely to be broken upon the mixing tables. In order to reduce the number of breakages, return the glassware to the lockers as soon as possible, and do not allow it to remain upon the table when not in use.

4. The student should also keep his desk and apparatus clean. At best a cement laboratory is a difficult place to keep clean on account of the cement dust, but a little care on the part of all of the students will greatly reduce the annoyance from this source. To remove cement which has set from glassware, vinegar or muriatic acid can be used. However, if glassware is thoroughly cleaned with water before the cement hardens, there will be no necessity for the use of acid.

5. **Assignment of Equipment.** Each student will be assigned a towel, a pan, a beaker, and a graduated cylinder. He will also be assigned a locker in which to keep his own equipment. The number of the locker will be the same as the reference number of the student, by which assignments will be made. Other equipment which will be required will be kept in lockers provided for

that purpose. Each of these lockers will contain the equipment other than that with which each student is provided, which is required for one problem. The keys for these lockers will be kept in the key case, and can be obtained at the beginning of each laboratory period. Whenever a key is taken from its hook in the key case a receipt card must be filled out and placed upon the hook. Upon the card should be placed the date, the number of the key or locker, and the name of the person taking the key. This card is not to be removed when the key is returned, but is to be allowed to remain as a record. After returning the equipment to the locker, the key is to be dropped through the slot in the key case provided for that purpose. In case any of the equipment is damaged, a red tag must be filled out and dropped into the key case with the key. Upon such red tags record the date, the article damaged, the locker in which the apparatus belongs, and the name of the person responsible for the damage. If apparatus is found to be damaged when the locker is assigned, report the facts at once to the instructor in charge, in order that the responsibility for the injury may be determined.

6. Molds for making briquettes, set of dies for marking test specimens, rammers, oil cans, waste, and other equipment and supplies of this character, will be kept in suitable places provided for them. Whenever such equipment is used, it should be

returned to its proper place as soon as possible, in order that the work of others may not be unnecessarily delayed.

7. Assignment of Materials. Each student will be assigned the cements which are to be used at each laboratory period. These assignments will be made from two particular brands, one of which will be a portland cement, and the other either a natural or a pozzulona cement. For convenience each brand of cement will be given a number by which it will be designated upon the assignment sheet, but in reporting the results of problems the *name as well as the number* of the cement should be reported in every case. The assignment sheet will indicate whether one or both of the cements are to be used at one laboratory period. If desirable the same problem may be assigned for two different laboratory periods, only one cement being used at each period. Different kinds of sand will also be designated by number. Numbers from 1 to 49 inclusive will be reserved for portland cements, numbers from 50 to 79 inclusive for natural cements, numbers from 80 to 89 inclusive for pozzulona cements, and numbers from 90 to 99 inclusive for different kinds of sand.

8. Waste Materials. After using cement do not attempt to return it to the original bins or cans, even though it has not been mixed, but place it in the waste can provided for that purpose. If the cement is allowed to be returned to the original

supply, mistakes are likely to occur, and the various brands will soon become mixed. Do not place any unused mortar or other waste materials in the cans provided for unused cement. Waste boxes will be provided in which unused mortar, broken briquettes, and other wastes which are not liquid, can be deposited. Very wet or liquid wastes are to be placed in the waste jar provided for them, and *under no conditions are wastes or water containing cement to be placed in the sink.*

9. Assignment of Problems. In order that an excessive amount of equipment may not be required, the problems will be assigned in such a manner that only a few students will be working on each problem at any one laboratory period. When more than one problem is to be executed at one laboratory period, the problems will be given on the assignment sheet in the order in which they are to be executed. It is important that the student shall observe the order in which the problems are to be done, particularly for assignments including the determination of the time of set, in which case the specimens should be prepared as quickly as possible at the beginning of the period, and while waiting for the setting to occur another problem can be executed. The letter *m* is used upon the assignment sheet to indicate the laboratory periods at which specimens are to be made, and the letter *t* to indicate the periods at which specimens are to be tested.

10. Marking Test Specimens. In some of the problems instructions are given for marking specimens which are to be tested at some future laboratory period. The student should be careful to follow these instructions, since it is desirable to have all the members of the class use a uniform method in order that the attendant who cares for the specimens may know where they belong.

ART. 2. PREPARATION OF REPORTS.

11. A report is to be made out for each problem and is to be handed in within three days after the completion of the problem in the laboratory. Each problem is to be reported upon a separate sheet, even though the laboratory work for several problems may be executed at one laboratory period. It is also desirable to have the report for each problem occupy but one page. In order that reports may be uniform, it is essential that each student shall use only the kind of paper designated for such reports, and the use of other kinds of paper will be sufficient cause for the rejection of reports.

12. In addition to the reports to be submitted for each problem, the student will be required to submit a final report upon each of the two cements used. Each of these final reports shall contain a summary of the results of the tests which have been made upon the brand of cement under consideration, and in conclusion a statement shall be made concerning the character of the cement as indicated

by the results of the various tests. In this statement name the kinds of work for which the cement would be satisfactory and the kinds of work for which it might prove unsatisfactory. Also compare the results with the specifications recommended by the American Society for Testing Materials, which are given in Appendix II.

13. It is essential that both the reports of the problems and the final reports shall be carefully prepared. Forms for these reports are not given, for the reason that it is desired that students shall take the initiative and that they shall learn to devise reports. For much of the school work, forms of notes are furnished which show the exact way in which to record the various data and results obtained. Cement laboratory work may be different from the other work which the student has done, and many of the engineering problems for which he will have occasion to devise reports will undoubtedly be different from any of his previous work; but, if he will study the methods used in the arrangement of a few kinds of work, he should be able to apply those methods to other kinds of work. For this reason the student is asked to study carefully the arrangement of his reports. In devising forms for reports the following things should be remembered: A report should be terse, precise, complete, and neat. In order that it may be neat, it must be not only well arranged, but also legibly written or lettered. In order that a

report may be complete and at the same time terse, all of the necessary facts should be stated, but each fact should be stated as briefly as may be without detracting from the precision of the statement. It is well to tabulate all data and all results which can be expressed in such form. Similar kinds of facts should be collected and arranged under suitable headings, as "Object of Experiment," "Apparatus," "Method of Determination," "Data and Results," "Conclusions," etc. Such headings should be given sufficient prominence to enable anyone to see at once where the facts for which he is looking may be found. It is not advisable to use symbols in headings for tabulated data, as % for per cent. Also avoid the use of abbreviations in the title and other headings of the report.

CHAPTER II.

DESCRIPTION OF APPARATUS.

14. In order that the student may better understand the problems in the following chapter, some of the apparatus commonly found in cement-testing laboratories will be briefly described.

15. Cement Sampler. The device shown in Fig. 1 is used for obtaining samples for inspection from cement packed in barrels.

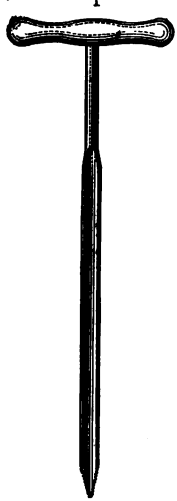

Fig. 1. — Cement Sampler

Fig. 2. — Single Sieve

Fig. 3. — Nest of Sieves with Cover and Pan

16. Sieves. Coarse sieves are used for screening cement to remove lumps, and fine sieves are used

for determining the fineness of cement. Both classes of sieves are designated by number, the number of meshes per lineal inch being the same as the number of the sieve. The styles of sieves ordinarily used in cement laboratories are shown in Fig. 2 and Fig. 3. Fig. 2 illustrates a single sieve, and Fig. 3 a nest of sieves arranged to fit together, with a cover on the top sieve and a pan attached to the lower sieve.

17. Apparatus for Determining Weight of Cement. For the purpose of determining the weight

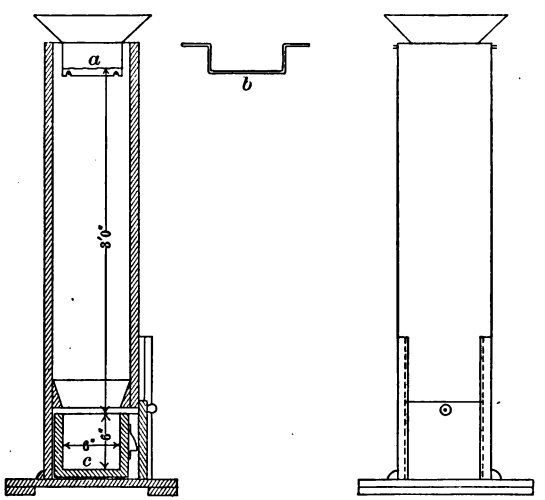

Fig. 4. — Apparatus for determining Weight of Cement

of cement per cubic foot or per bushel, the measuring box used must be so arranged that the amount

of compacting which the cement receives in entering the box shall be the same for the different samples tested, since the weight varies considerably with the degree of compactness. The device shown in Fig. 4 produces a uniform compactness by allowing the cement to fall from a coarse sieve *a*, suspended on hangers *b* to permit shaking, and fixed at a distance of three feet above the top of the measuring box *c*.

Fig. 5. — Sand-Glass

18. Sand-Glass. For the purpose of indicating the time of mixing to be used for making cement paste, a sand-glass, Fig. 5, is employed. Such glasses can be purchased, having a duration of flow of about two minutes.

19. Trowels. For use in cement laboratories pointing trowels, or masons' trowels, Fig. 6, are most useful. The most convenient sizes are 5-inch, 6-inch, and 10-inch trowels.

Fig. 6. — Mason's Trowel

20. Balances and Scales. For most of the weighing to be done in a cement laboratory the

Harvard Trip Balance, Fig. 7, or a scale with a scoop, Fig. 8, will be found satisfactory. The

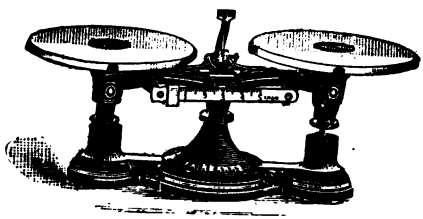

Fig. 7. — Harvard Trip Balance

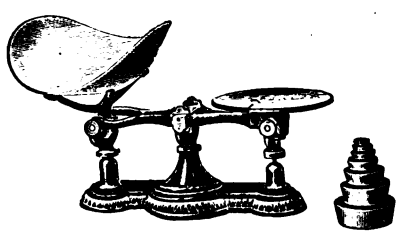

Fig. 8. — Scale for Cement and Sand

latter balance is probably a little better, since no time is required to balance the pan.

21. For use in making specific gravity determinations a balance of better quality should be used, but it is not necessary to use a fine balance of high grade.

22. For determining the fineness of cement a special scale, Fig. 9, is much used. This scale has a set of weights which have a unit such that 1000 units of cement will be a convenient quantity

for one determination, and thus each unit is equivalent to one-tenth of one per cent of the total.

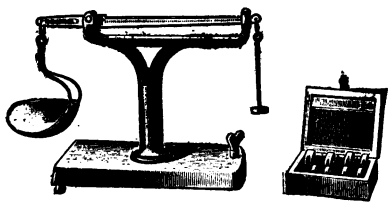

Fig. 9. — Scale for determining Fineness of Cement

23. Displacement Flasks. For the purpose of determining the specific gravity of cement, a flask

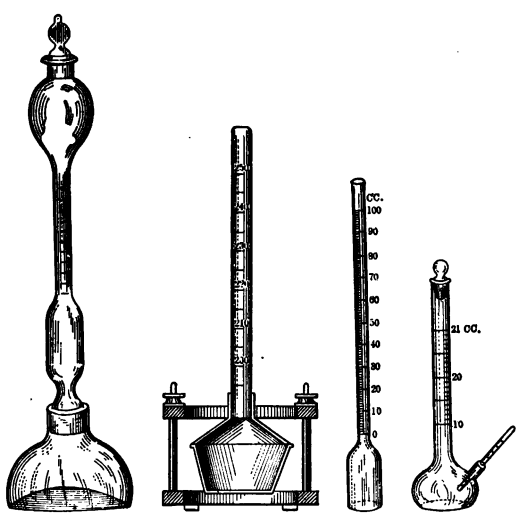

Fig. 10. — Displacement Flasks

is used which is graduated to indicate the volume displaced by the introduction of a small quantity

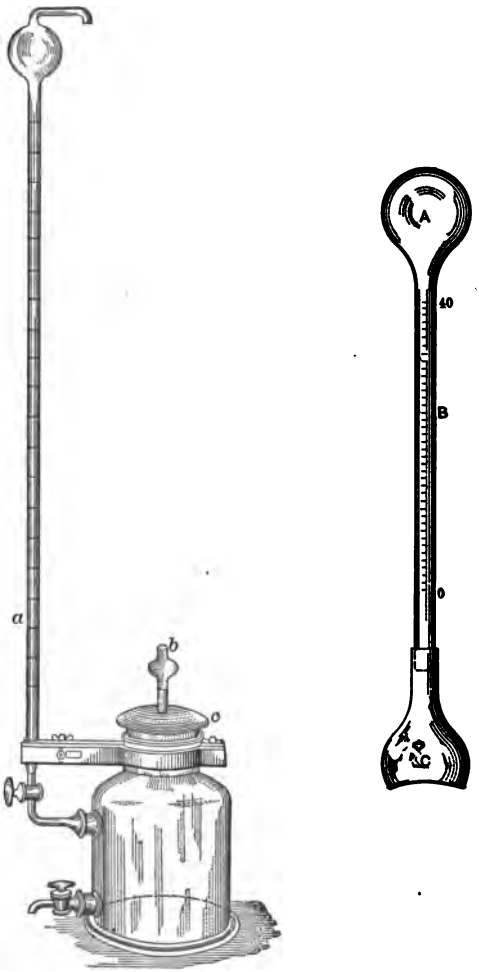

Fig. 10. — Displacement Flasks. — *Continued*

of cement into the liquid within the flask. Various types of flasks can be obtained from the makers, some of which are shown in Fig. 10. The apparatus recommended by the American Society of Civil Engineers is the Le Chatelier's flask, which when in use is to be immersed in a jar of water to reduce the amount of the variation in the temperature of the liquid within the flask. The Le Chatelier's apparatus is illustrated and described in Appendix I, page 95.

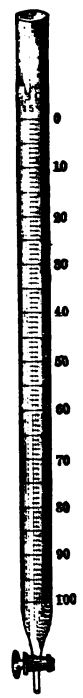

Fig. 12. — Burette

24. Measuring Glasses. For the purpose of measuring the quantity of water to be used in making cement paste or mortar, a graduated cylinder, Fig. 11, will be found useful. The sizes which will usually be of the most service are those having capacities of 100 c.c., 250 c.c., and 500 c.c. It is desirable to have both a small-sized and a large-sized cylinder at hand.

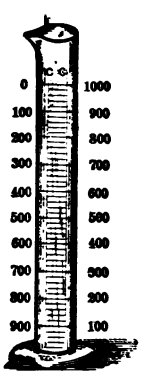

Fig. 11. — Graduated Cylinder

25. If water is piped to the mixing table a burette, Fig. 12, may be fastened beneath the tap and water measured in the burette.

26. Vicat Apparatus.* The Vicat apparatus,

* For a further description of the Vicat Apparatus, see Appendix I, page 98.

Fig. 13, is a device for measuring the distance which a weighted needle or weighted plunger will penetrate a ring filled with cement paste. There are two caps for each machine. The lighter cap is to be

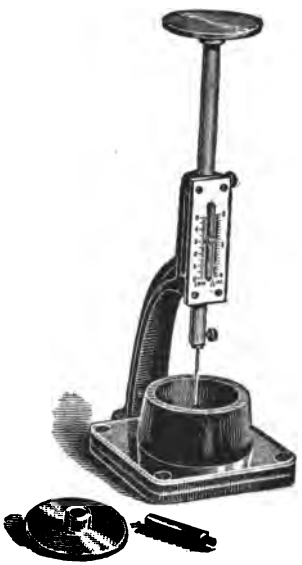

Fig. 13. — Vicat Apparatus

used only when the plunger is attached to the lower end of the piston, and the heavy cap is to be used only when the needle is in use. Care must be exercised to see that the proper cap is being used.

27. The Vicat apparatus is used for determining the proper percentage of water to be used in gauging the cement, and also for determining the rate

of setting. The plunger is employed for first purpose and the needle for the latter.

28. Gillmore's Needles. Another instrument for determining the time of setting of a sample of cement paste is Gillmore's needle, which consists of weighted wire or needle which is held in the hand and is brought to rest upon the surface of the paste. Two needles, Fig. 14, are required, one having a

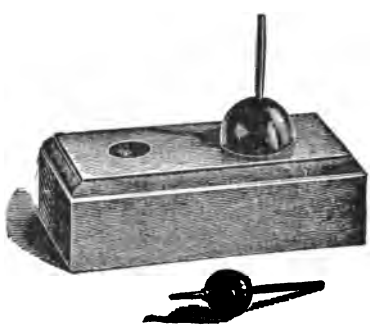

Fig. 14. — Gillmore's Needles

weight of one-fourth pound and a bearing area of one-twelfth inch, and one having a weight of one pound and a bearing area of one-twenty-fourth inch. The initial set is said to have taken place when a pat of cement will just support the light wire, and the final set when it will support the heavy wire.

29. Molds. The molds ordinarily employed in making specimens for determining the tensile

strength of cement mortar are of two kinds, individual molds and gang molds, both of which are illustrated in Fig. 15. The form of briquette

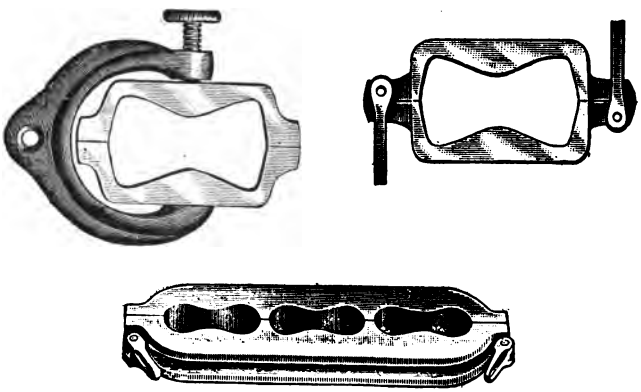

Fig. 15. — Briquette Molds

which is recommended by the American Society of Civil Engineers is shown in Fig. 25, page 104.

30. For the purpose of making specimens for compression tests of cement mortar, one-inch and two-inch cube molds, similar to the one shown in Fig. 16, are ordinarily used.

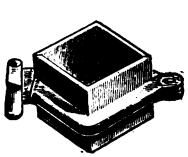

Fig. 16. — Cube Mold

31. **Molding Machines.** The most common method of molding briquettes is by hand, although machines for this purpose can be obtained from the manufacturer. Two machines of this kind are

the Olsen press, Fig. 17, and the Böhmé hammer, Fig. 18. With the Olsen press the briquette is molded by rotating the hand wheel which presses the mortar into a mold held in a clamp at the top of the machine. The amount of the pressure is recorded by the dial attached to the machine. With the Böhmé hammer the briquette is molded by compacting the mortar with a fixed number of blows of a hammer, the machine being arranged to stop automatically when the proper number of blows have been given.

32. The greatest value of briquette molding machines in a laboratory is to illustrate the effect of different methods of working cement mortar. The two machines illustrated in Figs. 17 and 18 are examples of the two classes into which all molding machines might be divided; viz., 1. Machines in which a steady pressure is employed, and 2. Machines in which the mortar is compacted by a blow. A comparison of the results obtained from two such machines is of value to a student to indicate the probable effect of different methods of manipulating cement in practical work.

33. **Boiler for Accelerated Tests.** For use in making boiling and steam tests a closed vessel is employed, having one or more racks upon which the specimens are placed. If the vessel is used for both boiling and steam tests, it is necessary to have two racks, one of which shall remain above the water level, and one below. The boiler recom-

Fig. 17. — Olsen Press

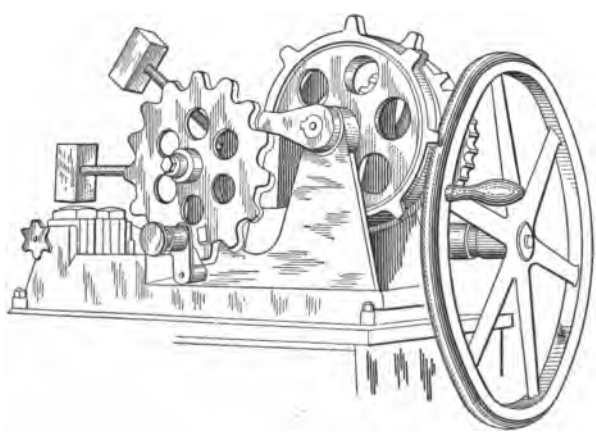

Fig. 18. — Böhmé Hammer

mended by the Committee on Uniform Tests of Cement of the American Society of Civil Engineers is shown in Fig. 28, page 109. This boiler is intended for steaming tests only, and therefore is provided with but one rack. This apparatus is arranged to maintain the level of the water three inches above the top of the boiler.

34. **Moist Closet.*** A moist closet is a box or chamber arranged so that the air within may be kept moist. It is used for the storage of test specimens from the time of making until they are immersed in water, usually twenty-four hours.

35. In some laboratories the test specimens are placed under a wet cloth instead of being placed in a moist chamber. The principal objection to this is that the cloth is likely to dry out, and is then of no service. To maintain the cloth in a moist condition the ends may be immersed in water, or a large pan may be turned upside down over the specimens and cloth. Of these two methods the writer prefers the latter, because he believes that it will produce a more nearly uniform condition for all of the specimens than will the first method. If a pan is used it should entirely cover all of the objects beneath, so that the entire rim of the pan may rest upon the table, thus preventing air currents from carrying away the moisture. If these precautions are observed, there will be no

* See paragraph 63, page 106.

difficulty in maintaining a moist condition of the cloth and air for twenty-four hours.

36. Storage Tank. Test specimens are usually stored in water from the time they are removed from the moist chamber until they are tested. For this purpose a storage tank is required which shall have a sufficient capacity to accommodate all of the specimens which are likely to be in storage at one time. For convenience in using such a tank should have a waste pipe, an overflow pipe leading to the waste pipe, and a supply pipe. If the tank is in a place which is open to the public, it is well to have the valves controlling the supply and waste pipes either locked or in a position such that they are not likely to be disturbed. It is also advisable to have a cover upon the tank which can be locked, so that specimens which are kept in storage for long-time tests may not be disturbed.

37. Testing Machines. For the purpose of determining the tensile or the compressive strength of test specimens a testing machine is used. A great variety of machines are upon the market of which three styles of machines for determining the tensile strength of cement are shown in Figs. 19, 20 and 21, and one type of machine for determining the compressive strength of small specimens is shown in Fig. 22. Some machines are arranged so that they may be used for both tensile and compressive tests. However, since very few compressive tests of cement are made, most of the testing

machines employed are for tensile tests. For testing concrete, compressive tests are often made, but for this purpose large testing machines are employed.

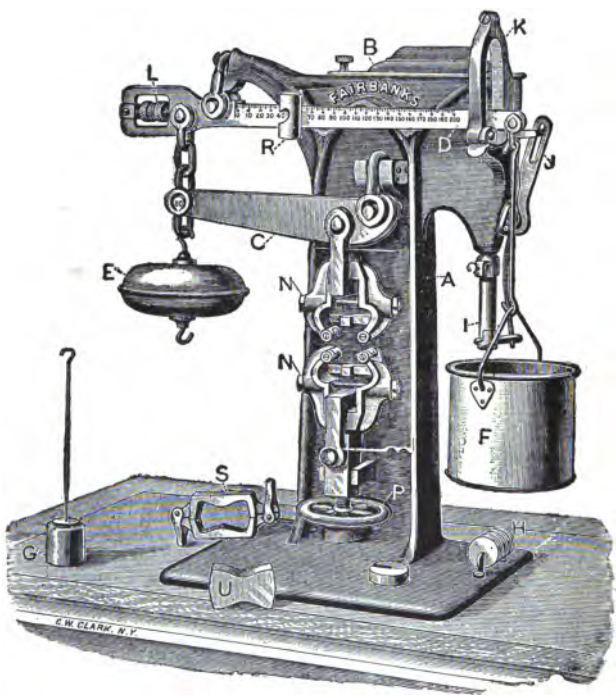

Fig. 19. — Automatic Testing Machine

38. The most common types of cement-testing machines produce a stress upon the briquette by means of a stream of shot which runs from a chamber of the machine into a pail or vessel.

When the briquette breaks, the stream of shot is automatically stopped, and the amount of the stress is determined by weighing or noting the weight of the shot in the vessel. The scale employed in the

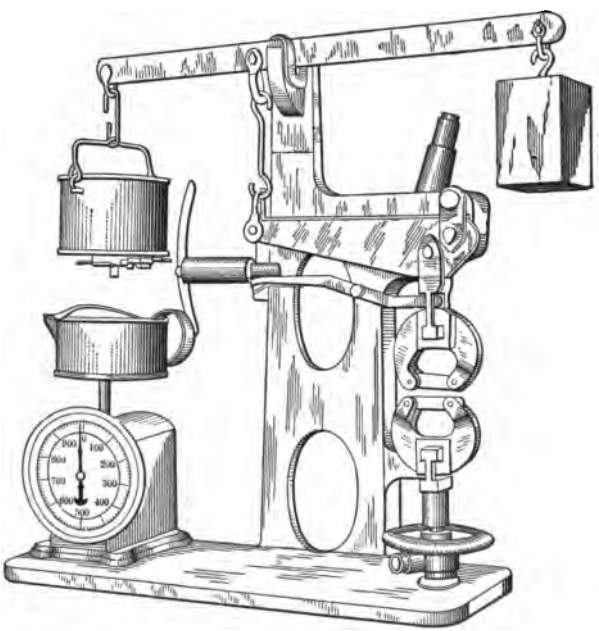

Fig. 20. — Automatic Testing Machine

weighing is so graduated that it indicates directly the stress upon the briquette, instead of the actual weight of the shot. The more improved forms of machines are arranged so that the beam of the machine can be kept horizontal while the stress is

being applied. If this cannot be done, it is usually necessary to estimate the amount of tension which must be applied to the briquette in placing it in the machine, in order that the beam may be nearly

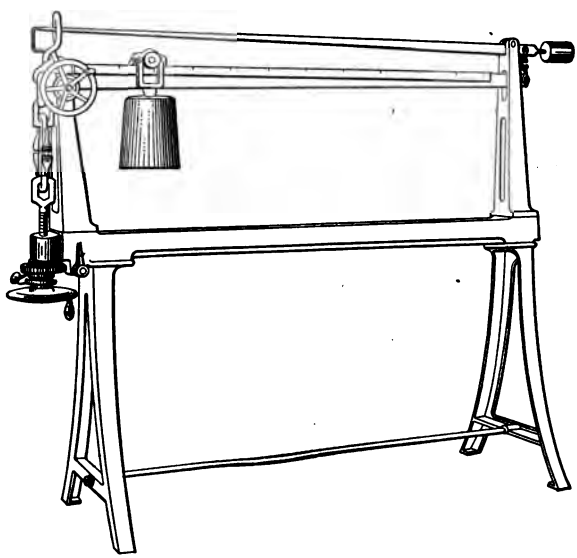

Fig. 21. — Beam Testing Machine

horizontal when the specimen breaks. The result is that weak specimens are often broken while being placed in the machine, and very strong specimens have to be reset in the machine, during which process they are likely to be broken.

39. The capacities which are usually employed for tensile-testing machines are 1000 and 2000

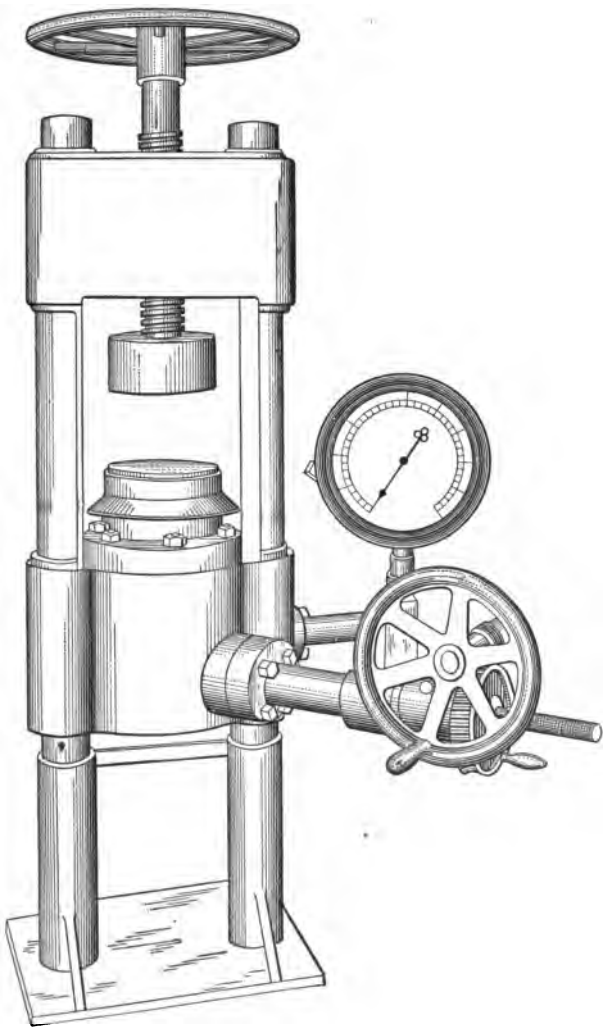

Fig. 22. — Machine for Compressive Tests

pounds. For ordinary laboratory work the lower capacity is probably the better, since very few briquettes will exceed 1000 pounds in strength, and since the lighter machine will act a little more quickly in shutting off the supply of shot. The hydraulic compression machine shown in Fig. 22 has a capacity of 40,000 pounds.

CHAPTER III.

LABORATORY PROBLEMS.

PROBLEM 1.

Determination of Fineness.

40. Apparatus Required. One set of sieves with cover and pan, scale for testing fineness, or a fine balance, with set of weights, apparatus for drying cement, and one No. 20 sieve.

41. Materials Required. 50 or 100 grams of each assigned cement.

42. Method of Operation. Thoroughly dry the cement, and then screen a small quantity through the No. 20 sieve, to remove the coarse lumps. Of the cement which passes through the sieve weigh out 1000 units if Riehle's scale for fineness is used, or 50 grams if a fine balance is used. Place the quantity thus weighed upon the No. 200 sieve, having the pan attached at the time. Place the cover upon the sieve and shake, holding the sieve in a slightly inclined position, and strike it gently with the palm of the hand at the rate of about 200 strokes per minute. Continue this operation until not more than one-tenth of one per cent passes through during one minute of continuous sieving.

Then weigh the residue retained upon the sieve and also the material in the pan. Next attach the No. 100 sieve to the pan, and place upon the sieve the residue caught upon the No. 200 sieve. The sieving is then continued in the same manner as with the No. 200 sieve. After completing the operation with this sieve the residue is placed upon the next coarser sieve, and so on until all of the sieves in the set have been used.

43. After completing the determination for one of the assigned cements determine the fineness of the other brand in the same manner.

44. Report. In the report state the quantities retained and the quantities passing each sieve. Also report the per cent of the total which is retained upon each sieve and the per cent which passes, stating the results to the nearest tenth of one per cent. Compare the results with the specification in Appendix II. Also note the errors of the experiment.

PROBLEM 2.

Weight of Cement.

45. Apparatus Required. Standard sifting box.*

46. Materials Required. About one-half peck of each assigned cement.

47. Method of Operation. Place a small quantity of cement upon the sieve of the standard sifting box, and shake the sieve. Continue to add small quantities of cement and to shake the sieve until the box at the bottom is filled. When the measuring box is full remove it carefully, and strike the top of the cement level with the sides of the box by means of a straight edge. Then weigh the box together with the cement contained. Also weigh the box empty, and measure its inside dimensions.

48. Repeat the entire operation with the other assigned cement.

49. After using the cement for this experiment do not return it to the cans or bins from which it was taken, unless special instructions to that effect have been given, but place it in the waste-cement can.

50. Report. Compute and tabulate the weight in pounds per bushel and in pounds per cubic foot. Also state the capacity of the box.

* See Fig. 4, page 11.

PROBLEM 3.

*Specific Gravity.**

51. **Apparatus Required.** LeChatelier flask with jar and small funnel,† glass rod, pipette, thermometer, small scoop, balance and set of weights, ring stand or other similar support, and apparatus for drying cement.

52. **Materials Required.** 200 grams of each assigned cement, and about one quart of benzine.

53. **Method of Operation.** Place a small quantity of each assigned cement (75 grams) on an iron plate and dry thoroughly. While these samples are drying determine the specific gravity of each cement in its undried condition.

54. Place enough water in the jar of the LeChatelier apparatus to half fill it. Insert enough benzine ‡ into the flask to bring the surface just a little above the mark below the small bulb. Place the flask in the jar of water and allow the benzine and water to attain the same temperature. Arrange the ring stand so that the flask will be held vertically in the jar of water and so that the small funnel will extend about an inch into the top of the

* For a discussion of the usefulness of the specific gravity test, see *The Specific Gravity of Portland Cement*, by Richard K. Meade and Lester C. Hawk, Proc. Am. Soc. for Testing Materials, vol. VII, p. 363.

† See Fig. 23, page 95.

‡ See paragraph 11, page 96.

flask. By means of the pipette withdraw enough benzine to bring the surface exactly to the mark below the small bulb. Weigh out 65 grams of cement, and insert it into the flask through the funnel, a little at a time, first noting the temperature of the water. A little of the cement can be taken in the scoop and can be passed through the funnel by using the glass rod. It is necessary to introduce only a small quantity of cement at a time to prevent clogging the stem of the flask near the surface of the benzine, and also to prevent air bubbles from being carried into the liquid with the cement. When all of the cement has been inserted in the flask read the position of the surface of the benzine and note the temperature of the water.

55. The volume between the mark below the small bulb and the zero of the graduation above the bulb is 20 c.c. The units of the graduation are cubic centimeters and the smallest divisions are tenths of centimeters. The specific gravity of the cement will be 65 divided by the volume displaced. If the temperature of the benzine has changed during the determination, a correction to the apparent displaced volume can be obtained by multiplying the total volume of benzine used, by the number of degrees change in temperature, by the coefficient of expansion of benzine. (Benzine is not a very definite compound, but for the purpose of making this correction it will be sufficiently exact to use 0.0014 as the coefficient of expansion per

degree centigrade.) The correction will be subtracted from the observed value of the displaced volume for an increase in temperature, and will be added for a decrease in temperature.

56. Next determine the specific gravity of each dried cement, following the instructions given in paragraph 54, and being careful to cool the cement to the temperature of the liquid before inserting it into the flask.

57. To prepare the LeChatelier flask for the second determination, pour into the bottle marked "Used Benzine" enough of the liquid to lower the surface to a little above the mark below the small bulb, and then bring the surface exactly to the mark by means of the pipette. After completing the second determination, carefully pour into the bottle marked "Used Benzine" as much of the liquid as possible until the cement begins to pass out. Then shake the flask over the waste jar to remove the remainder of the cement and benzine. Complete the cleaning by placing a little water in the flask and shaking it into the waste jar. Rinse two or three times to remove every particle of cement.

58. Be careful not to break the flask, as it is expensive. If the flask should be broken, do not throw away the pieces until you find whether it can be repaired, since in the latter case the charge for the breakage will probably be much less than the cost of a new flask.

59. Instead of determining the volume of a known weight of cement by inserting all of the 65 grams, the method of finding the weight of a given volume could have been used by inserting just enough cement to displace some given volume, say 20 c.c., and by reweighing the remainder of the cement. In this case the specific gravity would have been computed by dividing the weight of the cement introduced by 20.

60. **Report.** In the report for this problem state the apparatus by which each result was obtained. State the difficulties encountered and the precautions which should be taken. State whether the specific gravity of the liquid used affects the results, and state whether water, alcohol, kerosene, gasoline or turpentine could be used instead of benzine. Report your results to the nearest hundredth and compare your results with the specification in Appendix II.

PROBLEM 4.

Plasticity—Boulogne Method.

61. Apparatus Required. Trowel, pan, beaker, graduated cylinder, and coarse balance with set of weights.

62. Materials Required. 1000 to 1500 grams of each assigned cement, and water for mixing.

63. Method of Operation. Weigh out 500 grams of cement, place it upon the mixing table, form a crater at the center of the pile, pour into the crater a known quantity of water (say 21 per cent of the weight for portland cements, and 30 per cent for natural cements), turn the cement into the crater from the edges of the pile, and work the paste vigorously with a trowel for about five minutes. To determine whether the paste is of the proper consistency, apply the following tests: 1. The consistency of the paste should not change if gauged for an additional period of three minutes after the initial five minutes. 2. A small quantity of paste dropped from the trowel upon the mixing table from a height of 0.50 meter (20 inches) should leave the trowel clean, and should retain its form approximately without cracking. 3. A small quantity of paste worked gently in the hands should be easily molded into a ball, on the surface of which water should appear. When this ball is dropped from a height of 0.50 meter (20 inches), it should

retain a rounded form without cracking. 4. If a slightly smaller quantity of water be used the paste should be crumbly and should crack when dropped upon the table. On the other hand, the addition of a greater quantity of water — one or two per cent — should soften the paste, rendering it more sticky, and preventing it from retaining its form when dropped upon the table.

64. If the paste is too dry to fulfill the requirements of the tests described, add a little more water, carefully noting the amount, and repeat the tests. When the correct consistency is obtained, try a fresh sample to check the result, since some error may arise from adding water to paste which has been mixed for several minutes.

65. Report. In reporting this problem tabulate the results for each trial, stating the character of the paste obtained with each percentage of water. Also record the trials made with each mixture, showing the number of mixtures for each brand of cement. State the estimated error in the results as indicated by the quantity of water required to produce a noticeable change in the consistency of the paste.

PROBLEM 5.

Plasticity — with Vicat Apparatus.

66. Apparatus Required. Vicat machine fitted with plunger and with cap marked "Piston"; 1 vulcanite ring; 1 plate of glass about 4 inches by 4 inches; sand-glass, and the apparatus required for Problem 4.

67. Materials Required. 2000 to 2500 grams of each assigned cement, and water for mixing.

68. Method of Operation. Determine the per cent of water required to make a plastic paste for each assigned cement by the Tetmajer method and by the method recommended by the American Society of Civil Engineers.

69. Tetmajer Method. Mix 500 grams of cement into a paste, following the method described in Problem 4, noting the per cent of water used. Place the ring of the Vicat apparatus (large end up) on a piece of plate glass, fill with the paste, and carefully smooth off the top. See that the piston of the Vicat apparatus works smoothly. Then note the reading on the scale when the plunger rests upon the surface of the glass, outside of the ring. Next bring the plunger to rest upon the surface of the paste, then suddenly release it and allow the piston to descend of its own weight. When it finally comes to rest, note the reading on the scale. The paste is of the proper consistency if the plunger

comes to rest six millimeters above the glass. If the paste is too dry add a small quantity of water, and repeat the determinations until the proper consistency is obtained. Then check the result with a fresh mixture.

70. American Society of Civil Engineers' Method. Weigh out 500 grams of cement, place it upon the mixing table, form a crater at the center of the pile, pour into the crater a known quantity of water, turn the dry material from the edge of the pile into the crater with a trowel, and allow the material to stand until the water has been absorbed. Complete the mixing by vigorously kneading the paste with the hands for one and one-half minutes, the process being similar to that used in kneading dough. (The sand-glass is used to indicate the time of kneading.) Form the paste quickly into a ball with the hands, completing the operation by tossing it six times from one hand to the other, with the hands about six inches apart. Then press the ball into the ring through the larger opening. Place the ring upon the glass with the small end up, and smooth the surface of the paste to a level with the top of the ring. Bring the plunger of the Vicat machine to the surface of the paste, noting the reading on the scale, and release the plunger quickly. When the plunger finally comes to rest, note the reading on the scale. The paste is of the proper consistency if the piston comes to rest 10 millimeters below the top of the ring. Repeat the trials

in a similar manner, using a fresh mixture each time, until a paste is obtained which has the proper consistency.

71. **Report.** In reporting this problem follow the instructions for Problem 4, and in addition to the results for Problem 5 state the final results for Problem 4, for comparison. Also plot the results for Problem 5 upon coördinate paper, using percentage of water as abscissas and depth of penetration as ordinates. Draw a curve for the plotted values for each brand of cement.

PROBLEM 6.

Soundness — Cold-Pat Test

72. **Apparatus Required.** Trowel, pan, beaker, graduated cylinder, sand-glass, coarse balance with set of weights, and 8 pieces of glass each about 3 inches by 3 inches.

73. **Materials Required.** 500 grams of each assigned cement, and water for mixing.

74. **Method of Operation.** Mix 500 grams of one of the assigned cements into a plastic paste, using the per cent of water obtained in Problem 5 by the method recommended by the American Society of Civil Engineers. Make four circular pats, each having a diameter of about 3 inches and a thickness at the center of about one-half inch. The edges of the pat should be made as thin as possible. Stamp each pat with the number of the cement and with the number of your locker. Place the pats in the moist chamber, from which they will be removed at the end of twenty-four hours, and will be placed in pans having the same number as the locker. Fill one of these pans with water so that when removed from the moist chamber two of the four pats will be placed in air and two in water.

75. Make four similar pats, using the other assigned brand of cement, and place them in the moist chamber beside the first four.

76. Examine the pats each week for four successive weeks, at the end of which time the problem is to be reported.

77. Report. In the report of this problem state the condition in which each pat was found at each examination. State whether the pats loosened from the glass, whether the pats in water appeared green, whether the pats in air developed brown spots, whether the glass was cracked by the pats in water, whether radial cracks developed near the edges of the pats, and whether the cement disintegrated. Also note any other peculiar conditions. Consult your text-books to find the causes which are likely to produce the results mentioned, and state your opinion of the soundness of the cements tested.

PROBLEM 7.

Soundness — Accelerated Test.

78. Apparatus Required. Closed vessel arranged to boil water, provided with one rack supported in the water upon which to place specimens for the boiling test, and one rack above the water level on which to place specimens to be subjected to steam bath; also the apparatus required for Problem 6.

79. Materials Required. 500 grams of each assigned cement, and water for mixing.

80. Method of Operation. Make and mark four pats of each assigned cement, following the instructions for Problem 6. Place all of the pats in the moist chamber, and allow them to remain there until the boiling and steam tests are made, which should be at the end of twenty-four hours, but which may have to be deferred until the next laboratory period. (The time at which the problem is to be completed is indicated upon the assignment sheet.) To complete the test, place enough water in the boiler to bring the surface midway between the two racks. Place two pats of each cement in the water, and two pats of each cement on the rack above the water. Place the cover upon the boiler, and apply heat so as to raise the temperature of the water to the boiling-point in about thirty minutes. Continue the boiling until

the end of the laboratory period, provided the period of boiling does not exceed three hours. Do not remove the specimens from the boiler within an hour after the application of heat is discontinued. When the pats are removed, examine them carefully for indications of unsoundness.

81. Report. In reporting this problem follow the instructions given for Problem 6; and if Problem 6 has been completed, compare the results of the two problems.

PROBLEM 8.

Time of Setting.

82. Apparatus Required. Trowel, pan, beaker, graduated cylinder, Vicat apparatus with needle, two vulcanite rings, set of Gillmore wires, thermometer, sand-glass, 6 plates of glass about 3 inches by 3 inches, moist chamber, and pan of water.

83. Materials Required. 600 grams of each assigned cement, and water for mixing.

84. Method of Operation. Weigh out 600 grams of cement, and mix into a plastic paste, using the per cent of water obtained in Problem 5 by the method recommended by the American Society of Civil Engineers. Use water having a temperature about that of the air, and record its temperature. Fill one of the rings in the same manner as in determining the per cent of water. Form the remainder of the paste into three pats. Place one of the pats in the moist chamber, one in water, and allow one to harden in air. Note the temperature of the air.

85. In the same manner as above described, make a plastic paste of the other assigned cement, fill the second vulcanite ring, and make three pats. Allow these specimens to harden under the same conditions as the first set.

86. Examine each specimen at intervals of, say, ten minutes to see when setting begins and when it is

completed. Mix the cement promptly at the beginning of the laboratory period to allow as much time as possible for setting during the period.

87. The Gillmore wires are used with the pats. When the light wire is just supported upon the surface the initial set is said to have occurred, and when the heavy wire is supported the final set is said to have occurred. Note the mark made by the thumb nail at the time of the initial set and of the final set.

88. The Vicat apparatus is used with the rings. To test one of the specimens, bring the needle to the surface of the paste and release it suddenly. The initial set is said to have occurred when the needle ceases to pass a point five millimeters above the bottom of the ring. The final set is said to have occurred when the needle ceases to penetrate the surface. (Note that the Vicat apparatus is used only with the rings, and that the Gillmore wires are used only with the pats. The two methods of determining the points at which the initial and final sets occur are arbitrary, and hence the results of the two methods need not agree.) In using the Vicat apparatus keep the needle clean and see that the slide works freely.

89. Report. In reporting this problem compare the results by the two methods, state conditions affecting the rate of setting in each case, and state what errors are likely to occur.

PROBLEM 9.

Tensile Strength of Neat Cement — Variation with Age.

90. Apparatus Required. Trowel, pan, beaker, graduated cylinder, No. 20 sieve, sand-glass, thermometer, individual or gang molds for twenty briquettes, and coarse balance with set of weights.

91. Materials Required. 1500 grams of each assigned cement, and water for mixing.

92. Method of Operation. If the molds are not already cleaned and oiled, clean them by scraping off as much mortar as possible with a trowel and by removing the remainder with a wire brush. Then oil the molds with an oily rag, and also oil the top of the mixing table where the molds are to be placed. Be careful to oil the entire surface of the molds, particularly where they come in contact with the briquettes, but be careful not to use an excess of oil, since it may be injurious to the strength of the cement.

93. Screen the cement to remove the lumps, and then weigh out 1500 grams. Mix the cement into a paste, using the per cent of water which was determined in Problem 5 to produce the consistency recommended by the American Society of Civil Engineers. Record the per cent of water, the temperature of the water, and the temperature of the air. (The temperature of the water and of the air should

be about 70° F. or 21° C.) Mold ten briquettes of the paste, using the method recommended by the American Society of Civil Engineers, which is given in Appendix I. Note the interval elapsing from the time of adding water until the last briquette is molded. In like manner mix the other cement and mold ten more briquettes. Mark each briquette with the number of the cement and with your own number. Do not stamp the briquette near the breaking section.

94. Cover all of the briquettes with a damp cloth, and then turn a large pan upside down over them to keep them from drying out. (If a moist chamber is provided for the purpose, place the molds in the moist chamber instead of covering as directed above.) At the end of twenty-four hours the briquettes will be removed from the molds and placed in water in the storage tanks.

95. By means of a testing machine determine the tensile strength of five briquettes of each cement at the age of seven days, and of the remaining briquettes at twenty-eight days. If a shot machine is used, time the rate of flow for half a minute. If necessary adjust the rate of flow so that the force will be applied at the rate of 600 pounds per minute. Be careful to center each briquette properly in the grips, and see that the bearing surfaces of the grips are free from dirt and sand.

96. Report. In the report for this problem tabulate the results, giving the per cent of water for

each mixture, the time required for molding each set, the temperature of the water and the air, the brand of cement used, the age of each briquette when broken, the rate of application of the breaking load, the tensile strength of each briquette, the mean for each age, and the probable error for each mean as determined by the formula $Em = 0.6745 \sqrt{\frac{\Sigma d^2}{n(n-1)}}$, in which Em is the probable error, d is the difference between any result and the mean for the set to which it belongs, and n is the number of briquettes whose results are used in computing the mean. (Note that Em is in the same units as the results, and is not per cent of error.)

97. Plot the results of the mean for each age upon coördinate paper, and connect the points by a broken line. Use the ages at which the briquettes were broken as abscissas, and the breaking strengths as ordinates. Also compare the results with the specifications in Appendix II.

PROBLEM 10.

Tensile Strength of 1:3 *Mortar — Variation with Age.*

98. **Apparatus Required.** All of the apparatus required for Problem 9.

99. **Materials Required.** 400 grams of each assigned cement, 2400 grams of sand, and water for mixing.

100. **Method of Operation.** Prepare the molds as directed in Problem 9. Screen the cement through the No. 20 sieve, and weigh out 400 grams. Also weigh out 1200 grams of sand. Mix the cement and sand together dry, form a crater at the center of the pile, and pour into the crater the amount of water indicated by the table in Appendix I. Then mix into a mortar in the manner directed in Appendix I. From this mortar make and mark ten briquettes, following the directions for Problem 9. In like manner make and mark ten briquettes, using the other assigned cement. Store and test the specimens as directed in Problem 9.

101. **Report.** In the report for this problem follow the instructions given in Problem 9.

PROBLEM 11.

Variation in Tensile Strength of Neat Cement with Amount of Water.

102. Apparatus Required. Trowel, pan, beaker, graduated cylinder, individual or gang molds for sixteen briquettes, No. 20 sieve, sand-glass, thermometer, and coarse balance with set of weights.

103. Materials Required. 2400 grams of the assigned cement, and water for mixing.

104. Method of Operation. Prepare all of the molds as directed in Problem 9. Screen all of the cement through the No. 20 sieve and weigh out 600 grams. Mix the cement into a plastic paste, using the per cent of water which was determined in Problem 5 to be required for the consistency recommended by the American Society of Civil Engineers. Record the per cent of water, and the temperature of the water and of the air. (The temperature should be about 70° F. or 21° C.) Mold four briquettes of the paste, using the method given in Appendix I. Record the interval elapsing from the time of adding water until the last briquette is molded. Mark each briquette with your own number, and with the letter *P* to indicate that they were made with plastic paste.

105. Next weigh out 600 grams of the screened cement and mix it with water, using three per cent less water than was required for the plastic paste.

Mold four briquettes from this paste, using the same method as was used for the first set. Mark the briquettes with your number, and with the letter *D* to indicate that they were made with a dry paste.

106. In like manner make four briquettes, using six per cent less water than was used for the plastic paste. Mark these briquettes with your number, and with the letters *VD* to indicate that they were made with very dry paste.

107. In like manner make four briquettes, using three per cent more water than was used for the plastic paste. Mark these briquettes with your number, and with the letter *W* to indicate that they were made with a wet paste.

108. Store the briquettes as described in paragraph 94, page 68.

109. When the briquettes are seven days old, or at the next laboratory period, remove them from the storage tank, and determine the tensile strength of each briquette by means of a testing machine. Observe the precautions given in paragraph 95, page 68.

110. Report. In the report for this problem tabulate the results, giving the per cent of water for each mixture, the time required for molding each set of briquettes, the temperature of the water and of the air, the brand of cement, the age at which the briquettes were broken, the rate of application of the breaking load, the tensile strength of each

briquette, the mean tensile strength for each set of briquettes, and the probable error* of the mean of each set.

111. Plot the results of the mean for each set of observations upon coördinate paper, and connect the points by a series of broken lines. Use percentages of water for abscissas, and breaking strengths for ordinates.

112. In conclusion state what is shown by your results concerning the effect of the amount of water upon the tensile strength.

* For formula for probable error, see paragraph 96, p. 70.

PROBLEM 12.

Tensile Strength of 1:3 *Mortar — Effect of Different Methods of Molding.*

113. **Apparatus Required.** Trowel, pan, beaker, graduated cylinder, individual molds for twelve briquettes, No. 20 sieve, sand-glass, thermometer, coarse balance with set of weights, Olsen briquette molding machine, and Böhmé hammer.

114. **Materials Required.** 500 grams of the assigned cement, 1500 grams of sand (be careful to use the sand which is assigned), and water for mixing.

115. **Method of Operation.** If the molds are not ready for use, clean and oil them as described in Problem 9. Also oil the table under the molds. Screen the cement through the No. 20 sieve to remove the lumps. Weigh out 500 grams of cement and 1500 grams of sand, and mix them together dry. Form a crater at the center of the pile, into which pour the amount of water required for the proper percentage as shown in the table on page 100. Complete the mixing by the method of the American Society of Civil Engineers as explained in Problem 5. Mold four briquettes by hand, four with the Olsen press, and four with the Böhmé hammer. For each machine-made briquette weigh out 175 grams of mortar. Mold the briquette promptly after mixing, and keep the mortar

covered with a damp cloth while molding the briquettes.

116. Record the per cent of water used, the temperature of the water and of the air, and the interval from the time of adding the water until beginning to mold the first briquette, and until the completion of molding the last briquette.

117. Mark the briquettes, using your own number and the letters *H*, *O*, and *B* for hand-made briquettes, Olsen-press briquettes, and Böhmé-hammer briquettes, respectively. Store the briquettes as directed in Problem 9, and break them at the age of seven days or at the next laboratory period.

118. Report. In the report for this problem tabulate the results, giving all of the observed data and results. State the tensile strength of each briquette, the mean for each set and the probable error * for each mean. Compare the strength of these briquettes with the strength of the neat cement briquettes made from the same brand of cement in Problem 9. Also compare the results of this problem with the specifications given in Appendix II.

* For formula for probable error, see paragraph 96, p. 70.

PROBLEM 13.

Comparison of Different Methods of Hand Molding.

119. **Apparatus Required.** Trowel, pan, beaker, graduated cylinder, individual or gang molds for sixteen briquettes, No. 20 sieve, sand-glass, thermometer, and coarse balance with set of weights.

120. **Materials Required.** 3000 grams of the assigned cement, and water for mixing.

121. **Method of Operation.** If the molds are not ready for use, clean and oil them as directed in Problem 9. Weigh out 1500 grams of screened cement and mix into a paste, using three per cent less water than was required for the plastic paste in Problem 5 by the method of the American Society of Civil Engineers. Make five briquettes in each of the following ways: 1. by entirely filling the molds, pressing the mortar into place with the thumbs, and troweling the surface; 2. by partially filling the mold and pressing the paste into place after the addition of each increment; 3. by partially filling the mold and ramming each increment with an oak rammer having a cross-section of about three-fourths inch by three-fourths inch, and having a length of twelve inches; 4. by using a half-inch round iron or brass rammer, about twelve inches long. (In using a rammer, the compression should be produced by the blow of rod, and not by pushing

the rammer into the paste.) Be careful to avoid injuring the edges of the molds.

122. Mark each briquette with your number, and in addition mark the first five with 1, the second five with 2, etc. Store the briquettes as directed in Problem 9, and break at the age of seven days or at the next laboratory period.

123. Record the per cent of water used, the temperature of the water and of the air, the time from adding the water until beginning to mold the first briquette, and until the completion of molding the last briquette for each mixture.

124. Report. In the report for this problem tabulate the results, giving all of the observed data. State the tensile strength of each briquette, the mean for each set, and the probable error * for each mean.

* For formula for probable error, see paragraph 96, p. 70.

PROBLEM 14.

Compressive Strength of Cement and Cement Mortar.

125. **Apparatus Required.** Trowel, pan, beaker, graduated cylinder, No. 20 sieve, sand-glass, thermometer, six molds for one-inch cubes, six molds for two-inch cubes, and coarse balance with set of weights.

126. **Materials Required.** 1000 grams of the assigned cement, 1200 grams of sand, and water for mixing.

127. **Method of Operation.** If the molds are not ready for use, clean and oil them as directed in Problem 9. Weigh out 600 grams of cement and mix into a paste, using the per cent of water determined in Problem 5 by the method of the American Society of Civil Engineers. Fill the one-inch molds with the paste.

128. Weigh out 400 grams of cement and 1200 grams of sand, and mix into a mortar as directed in Appendix I. Fill the two-inch molds, pressing the mortar into the molds with the thumbs.

129. Mark all of the specimens with your number, and store them as directed in Problem 9. Break three of the small cubes and three of the large cubes at the end of one week, and the others at the end of four weeks.

130. Report. In the report of this problem tabulate the data and the results, giving the strength of each specimen, the mean of each set for each age, and the probable error* of each mean. Compare the compressive strength with the tensile strength obtained in previous problems.

* For formula for probable error, see paragraph 96, p. 70.

APPENDICES

APPENDIX I.

PROGRESS REPORT OF COMMITTEE ON UNIFORM TESTS OF CEMENT OF THE AMERICAN SOCIETY OF CIVIL ENGINEERS.*

PRESENTED AT THE ANNUAL MEETING, JANUARY 21, 1903, *AMENDED AT THE ANNUAL MEETING, JANUARY* 20, 1904, *AND AT THE ANNUAL MEETING, JANUARY* 15, 1908.

SAMPLING.

1. SELECTION OF SAMPLE. The selection of the sample for testing is a detail that must be left to the discretion of the engineer; the number and the quantity to be taken from each package will depend largely on the importance of the work, the number of tests to be made, and the facilities for making them.

2. The sample shall be a fair average of the contents of the package; it is recommended that, where conditions permit, one barrel in every ten be sampled.

3. Samples should be passed through a sieve having twenty meshes per linear inch, in order to break up lumps and remove foreign material; this is also a very effective method for mixing them together in order to obtain an average. For determining the characteristics of a shipment of cement, the individual samples may be mixed and the average tested; where time will permit, however, it is recommended that they be tested separately.

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4. METHOD OF SAMPLING. Cement in barrels should be sampled through a hole made in the center of one of the staves, midway between the heads, or in the head, by means of an auger or a sampling iron similar to that used by sugar inspectors. If in bags, it should be taken from surface to center.

CHEMICAL ANALYSIS.

5. SIGNIFICANCE. Chemical analysis may render valuable service in the detection of adulteration of cement with considerable amounts of inert material, such as slag or ground limestone. It is of use, also, in determining whether certain constituents, believed to be harmful when in excess of a certain percentage, as magnesia and sulphuric anhydride, are present in inadmissible proportions.

6. The determination of the principal constituents of cement — silica, alumina, iron oxide, and lime — is not conclusive as an indication of quality. Faulty character of cement results more frequently from imperfect preparation of the raw material or defective burning than from incorrect proportions of the constituents. Cement made from very finely ground material, and thoroughly burned, may contain much more lime than the amount usually present and still be perfectly sound. On the other hand, cements low in lime may, on account of careless preparation of the raw material, be of dangerous character. Further, the ash of the fuel used in burning may so greatly modify the composition of the product as largely to destroy the significance of the results of analysis.

7. METHOD. As a method to be followed for the analysis of cement, that proposed by the Committee on Uniformity in the Analysis of Materials for the Portland Cement Industry, of the New York Section of the Society for Chemical Industry, and published in *Engineering News*, Vol. 50, page 60, 1903, and the *Engineering Record*, Vol. 48, page 49, 1903, is recommended.

Specific Gravity.

8. Significance. The specific gravity of cement is lowered by underburning, adulteration, and hydration, but the adulteration must be in considerable quantity to affect the results appreciably.

9. Inasmuch as the differences in specific gravity are usually very small, great care must be exercised in making the determination.

10. Apparatus and Method. The determination of specific gravity is most conveniently made with LeChatelier's

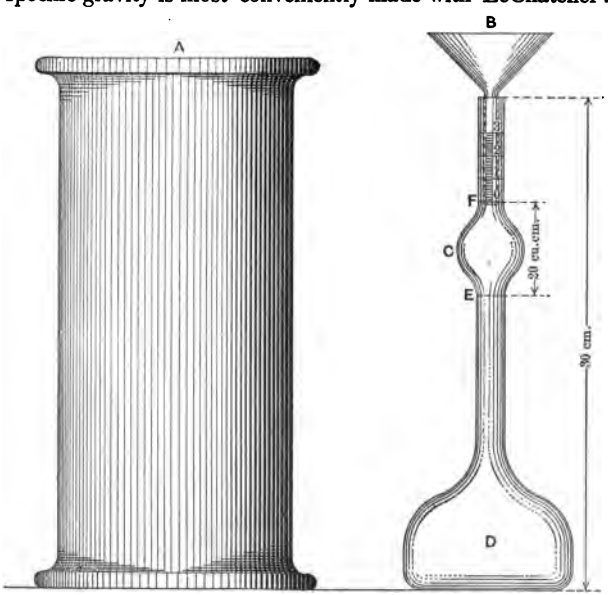

Fig. 23. — LeChatelier's Specific Gravity Apparatus

apparatus. This consists of a flask (*D*), Fig. 23, of 120 cu.cm. (7.32 cu. ins.) capacity, the neck of which is about 20 cm. (7.87 ins.) long; in the middle of this neck is a bulb (*C*),

above and below which are two marks (*F*) and (*E*); the volume between these marks is 20 cu. cm. (1.22 cu. ins.). The neck has a diameter of about 9 mm. (0.35 in.), and is graduated into tenths of cubic centimeters above the mark (*F*).

11. Benzine (62 degrees Bäumé naphtha), or kerosene free from water, should be used in making the determination.

12. The specific gravity can be determined in two ways:

(1) The flask is filled with either of these liquids to the lower mark (*E*), and 64 gr. (2.25 oz.) of powder, cooled to the temperature of the liquid, is gradually introduced through the funnel (*B*) [the stem of which extends into the flask to the top of the bulb (*C*)], until the upper mark (*F*) is reached. The difference in weight between the cement remaining and the original quantity (64 gr.) is the weight which has displaced 20 cu. cm.

13. (2) The whole quantity of the powder is introduced, and the level of the liquid rises to some division of the graduated neck. This reading plus 20 cu. cm. is the volume displaced by 64 gr. of the powder.

14. The specific gravity is then obtained from the formula:

$$\text{Specific Gravity} = \frac{\text{Weight of Cement, in grams}}{\text{Displaced Volume, in cubic centimeters}}.$$

15. The flask, during the operation, is kept immersed in water in a jar (*A*), in order to avoid variations in the temperature of the liquid. The results should agree within 0.01. The determination of specific gravity should be made on the cement as received; and should it fall below 3.10, a second determination should be made on the sample ignited at a low red heat.

16. A convenient method for cleaning the apparatus is as follows: The flask is inverted over a large vessel, preferably a glass jar, and shaken vertically until the liquid starts to flow freely; it is then held still in a vertical position until

empty; the remaining traces of cement can be removed in a similar manner by pouring into the flask a small quantity of clean liquid and repeating the operation.

17. More accurate determinations may be made with the picnometer.

FINENESS.

18. SIGNIFICANCE. It is generally accepted that the coarser particles in cement are practically inert, and it is only the extremely fine powder that possesses adhesive or cementing qualities. The more finely cement is pulverized, all other conditions being the same, the more sand it will carry and produce a mortar of a given strength.

19. The degree of final pulverization which the cement receives at the place of manufacture is ascertained by measuring the residue retained on certain sieves. Those known as the No. 100 and No. 200 sieves are recommended for this purpose.

20. APPARATUS. The sieves should be circular, about 20 cm. (7.87 ins.) in diameter, 6 cm. (2.36 ins.) high, and provided with a pan 5 cm. (1.97 ins.) deep, and a cover.

21. The wire cloth should be of brass wire having the following diameters:

No. 100, 0.0045 in.; No. 200, 0.0024 in.

22. This cloth should be mounted on the frames without distortion; the mesh should be regular in spacing and be within the following limits:

No. 100, 96 to 100 meshes to the linear inch.
No. 200, 188 to 200 meshes to the linear inch.

23. Fifty grams (1.76 oz.) or 100 gr. (3.52 oz.) should be used for the test, and dried at a temperature of 100° Cent. (212° Fahr.) prior to sieving.

24. METHOD. The Committee, after careful investigation, has reached the conclusion that mechanical sieving is

not as practicable or efficient as handwork, and, therefore, recommends the following method:

25. The thoroughly dried and coarsely screened sample is weighed and placed on the No. 200 sieve, which, with pan and cover attached, is held in one hand in a slightly inclined position, and moved forward and backward, at the same time striking the side gently with the palm of the other hand, at the rate of about 200 strokes per minute. The operation is continued until not more than one-tenth of 1 per cent passes through after one minute of continuous sieving. The residue is weighed, then placed on the No. 100 sieve and the operation repeated. The work may be expedited by placing in the sieve a small quantity of large steel shot. The results should be reported to the nearest tenth of 1 per cent.

Normal Consistency.

26. Significance. The use of a proper percentage of water in making the pastes * from which pats, tests of setting and briquettes are made, is exceedingly important, and affects vitally the results obtained.

27. The determination consists in measuring the amount of water required to reduce the cement to a given state of plasticity, or to what is usually designated the normal consistency.

28. Various methods have been proposed for making this determination, none of which has been found entirely satisfactory. The Committee recommends the following:

29. Method. Vicat Needle Apparatus. This consists of a frame (*K*), Fig. 24, bearing a movable rod (*L*), with the cap (*A*) at one end, and at the other the cylinder (*B*), 1 cm. (0.39 in.) in diameter, the cap, rod, and cylinder weighing 300 gr. (10.58 oz.). The rod, which can be held

* The term "paste" is used in this report to designate a mixture of cement and water, and the word "mortar" a mixture of cement, sand and water.

in any desired position by a screw (*F*), carries an indicator, which moves over a scale (graduated to centimeters) attached to the frame (*K*). The paste is held by a conical, hard-

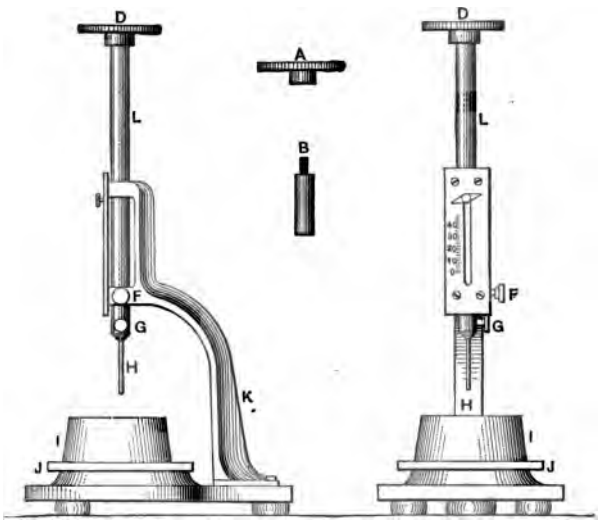

Fig. 24. — Vicat Needle

rubber ring (*I*), 7 cm. (2.76 ins.) in diameter at the base, 4 cm. (1.57 ins.) high, resting on a glass plate (*J*), about 10 cm. (3.94 ins.) square.

30. In making the determination, the same quantity of cement as will be subsequently used for each batch in making the briquettes (but not less than 500 grams) is kneaded into a paste, as described in paragraph 59, and quickly formed into a ball with the hands, completing the operation by tossing it six times from one hand to the other, maintained 6 ins. apart; the ball is then pressed into the rubber ring, through the larger opening, smoothed off, and placed (on its large end) on a glass plate and the smaller

end smoothed off with a trowel; the paste, confined in the ring, resting on the plate, is placed under the rod bearing the cylinder, which is brought in contact with the surface and quickly released.

31. The paste is of normal consistency when the cylinder penetrates to a point in the mass 10 mm. (0.39 in.) below the top of the ring. Great care must be taken to fill the ring exactly to the top.

32. The trial pastes are made with varying percentages of water until the correct consistency is obtained.

NOTE. The Committee on Standard Specifications for Cement inserts the following table for temporary use to be replaced by one to be devised by the Committee of the American Society of Civil Engineers.

PERCENTAGE OF WATER FOR STANDARD SAND MORTARS.

Neat.	One Cement Three Standard Ottawa Sand.	Neat.	One Cement Three Standard Ottawa Sand.	Neat.	One Cement Three Standard Ottawa Sand.
15	8.0	23	9.3	31	10.7
16	8.2	24	9.5	32	10.8
17	8.3	25	9.7	33	11.0
18	8.5	26	9.8	34	11.2
19	8.7	27	10.0	35	11.5
20	8.8	28	10.2	36	11.5
21	9.0	29	10.3	37	11.7
22	9.2	30	10.5	38	11.8

	1 to 1	1 to 2	1 to 3	1 to 4	1 to 5
Cement...	500	333	250	200	167
Sand.....	500	666	750	800	833

33. The Committee has recommended, as normal, a paste, the consistency of which is rather wet, because it believes that variations in the amount of compression to which the briquette is subjected in molding are likely to be less with such a paste.

34. Having determined in this manner the proper percentage of water required to produce a paste of normal consistency, the proper percentage required for the mortars is obtained from an empirical formula.

35. The Committee hopes to devise such a formula. The subject proves to be a very difficult one, and, although the Committee has given it much study, it is not yet prepared to make a definite recommendation.

Time of Setting.

36. Significance. The object of this test is to determine the time which elapsed from the moment water is added until the paste ceases to be fluid and plastic (called the "initial set"), and also the time required for it to acquire a certain degree of hardness (called the "final" or "hard set"). The former of these is the more important, since, with the commencement of setting, the process of crystallization or hardening is said to begin. As a disturbance of this process may produce a loss of strength, it is desirable to complete the operation of mixing and molding or incorporating the mortar into the work before the cement begins to set.

37. It is usual to measure arbitrarily the beginning and end of the setting by the penetration of weighted wires of given diameters.

38. Method. For this purpose the Vicat Needle, which has already been described in paragraph 29, should be used.

39. In making the test, a paste of normal consistency is molded and placed under the rod (*L*), Fig. 24, as described in paragraph 30; this rod, bearing the cap (*D*) at one end

and the needle (*H*), 1 mm. (0.039 in.) in diameter, at the other, weighing 300 gr. (10.58 oz.). The needle is then carefully brought in contact with the surface of the paste and quickly released.

40. The setting is said to have commenced when the needle ceases to pass a point 5 mm. (0.20 in.) above the upper surface of the glass plate, and is said to have terminated the moment the needle does not sink visibly into the mass.

41. The test pieces should be stored in moist air during the test; this is accomplished by placing them on a rack over water contained in a pan and covered with a damp cloth, the cloth to be kept away from them by means of a wire screen; or they may be stored in a moist box or closet.

42. Care should be taken to keep the needle clean, as the collection of cement on the sides of the needle retards the penetration, while cement on the point reduces the area and tends to increase the penetration.

43. The determination of the time of setting is only approximate, being materially affected by the temperature of the mixing water, the temperature and humidity of the air during the test, the percentage of water used, and the amount of molding the paste receives.

Standard Sand.

44. The Committee recognizes the grave objections to the standard quartz now generally used, especially on account of its high percentage of voids, the difficulty of compacting in the molds, and its lack of uniformity; it has spent much time in investigating the various natural sands which appeared to be available and suitable for use.

45. For the present, the Committee recommends the natural sand from Ottawa, Ill., screened to pass a sieve having 20 meshes per linear inch and retained on a sieve having 30 meshes per linear inch; the wires to have diameters of 0.0165 and 0.0112 in., respectively, *i.e.*, half the width of

the opening in each case. Sand having passed the No. 20 sieve shall be considered standard when not more than one per cent passes a No. 30 sieve after one minute continuous sifting of a 500-gram sample.*

Form of Briquette.

46. While the form of the briquette recommended by a former Committee of the Society is not wholly satisfactory, this Committee is not prepared to suggest any change, other than rounding off the corners by curves of ½-in. radius, Fig. 25.

Molds.

47. The molds should be made of brass, bronze, or some equally non-corrodible material, having sufficient metal in the sides to prevent spreading during molding.

48. Gang molds, which permit molding a number of briquettes at one time, are preferred by many to single molds, since the greater quantity of mortar that can be mixed tends to produce greater uniformity in the results. The type shown in Fig. 26 is recommended.

49. The molds should be wiped with an oily cloth before using.

Mixing.

50. All proportions should be stated by weight; the quantity of water to be used should be stated as a percentage of the dry material.

51. The metric system is recommended because of the convenient relation of the gram and the cubic centimeter.

52. The temperature of the room and the mixing water should be as near 21 degrees Cent. (70 degrees Fahr.) as it is practicable to maintain it.

* The Sandusky Portland Cement Company, of Sandusky, Ohio, has agreed to undertake the preparation of this sand, and to furnish it at a price only sufficient to cover the actual cost of preparation.

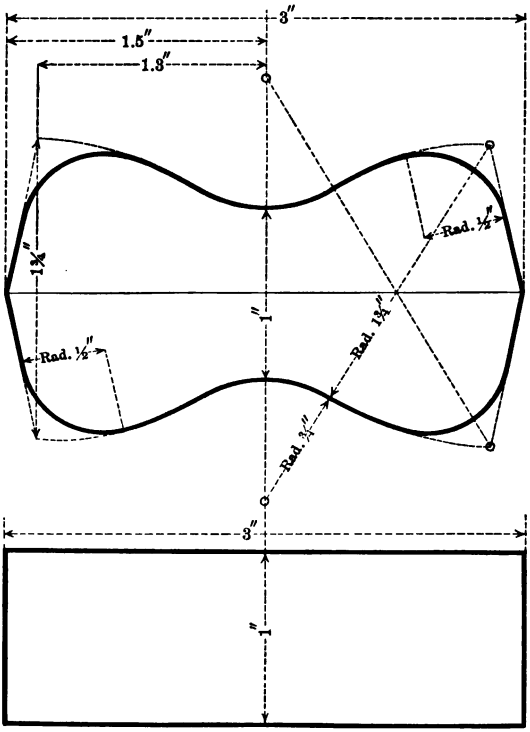

Fig. 25. — Details for Briquette

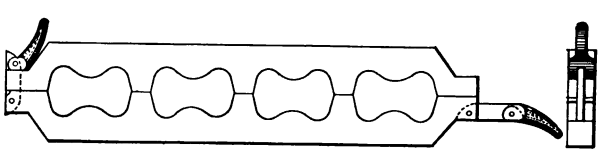

Fig. 26. — Details for Gang Mold

53. The sand and cement should be thoroughly mixed dry. The mixing should be done on some non-absorbing surface, preferably plate glass. If the mixing must be done on an absorbing surface, it should be thoroughly dampened prior to use.

54. The quantity of material to be mixed at one time depends on the number of test pieces to be made; about 1000 gr. (35.28 oz.) makes a convenient quantity to mix, especially by hand methods.

55. The Committee, after investigation of the various mechanical mixing machines, has decided not to recommend any machine that has thus far been devised, for the following reasons:

(1) The tendency of most cement is to "ball up" in the machine, thereby preventing the working of it into a homogeneous paste; (2) there are no means of ascertaining when the mixing is complete without stopping the machine; and (3) the difficulty of keeping the machine clean.

56. METHOD. The material is weighed and placed on the mixing table, and a crater formed in the center, into which the proper percentage of clean water is poured; the material on the outer edge is turned into the crater by the aid of a trowel. As soon as the water has been absorbed, which should not require more than one minute, the operation is completed by vigorously kneading with the hands for an additional 1½ minutes, the process being similar to that used in kneading dough. A sand-glass affords a convenient guide for the time of kneading. During the operation of mixing, the hands should be protected by gloves, preferably of rubber.

MOLDING.

57. Having worked the paste or mortar to the proper consistency, it is at once placed in the molds by hand.

58. The Committee has been unable to secure satisfactory results with the present molding machines; the operation of

machine molding is very slow, and the present types permit of molding but one briquette at a time, and are not practicable with the pastes or mortars herein recommended.

59. METHOD. The molds should be filled immediately after the mixing is completed, the material pressed in firmly with the fingers and smoothed off with a trowel without mechanical ramming. The mold should be turned over and the operation repeated.

60. A check upon the uniformity of the mixing and molding is afforded by weighing the briquettes just prior to immersion, or upon removal from the moist closet. Briquettes which vary in weight more than 3 per cent from the average should not be tested.

STORAGE OF THE TEST PIECES.

61. During the first 24 hours after molding, the test pieces should be kept in moist air to prevent them from drying out.

62. A moist closet or chamber is so easily devised that the use of the damp cloth should be abandoned if possible. Covering the test pieces with a damp cloth is objectionable, as commonly used, because the cloth may dry out unequally, and, in consequence, the test pieces are not all maintained under the same condition. Where a moist closet is not available, a cloth may be used and kept uniformly wet by immersing the ends in water. It should be kept from direct contact with the test pieces by means of a wire screen or some similar arrangement.

63. A moist closet consists of a soapstone or slate box, or a metal-lined wooden box — the metal lining being covered with felt and this felt kept wet. The bottom of the box is so constructed as to hold water, and the sides are provided with cleats for holding glass shelves on which to place the briquettes. Care should be taken to keep the air in the closet uniformly moist.

64. After 24 hours in moist air the test pieces for longer

periods of time should be immersed in water maintained as near 21° Cent. (70° Fahr.) as practicable; they may be stored in tanks or pans, which should be of non-corrodible material.

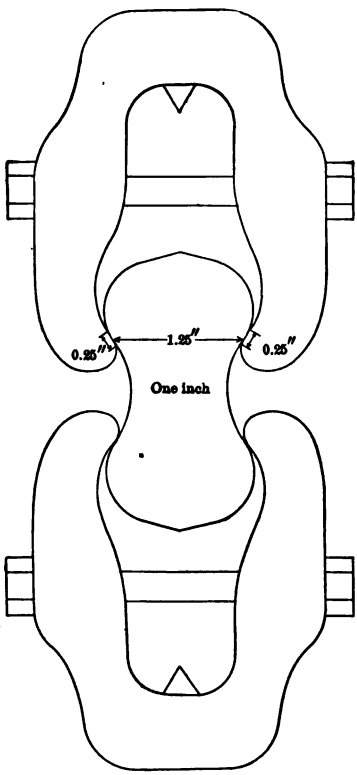

Fig. 27. — Form of Clip

TENSILE STRENGTH.

65. The tests may be made on any standard machine. A solid metal clip, as shown in Fig. 27, is recommended.

This clip is to be used without cushioning at the points of contact with the test specimen. The bearing at each point of contact should be $\frac{1}{4}$ in. wide, and the distance between the center of contact on the same clip should be $1\frac{1}{4}$ ins.

66. Test pieces should be broken as soon as they are removed from the water. Care should be observed in centering the briquettes in the testing machine, as cross-strains, produced by improper centering, tend to lower the breaking strength. The load should not be applied too suddenly, as it may produce vibration, the shock from which often breaks the briquette before the ultimate strength is reached. Care must be taken that the clips and the sides of the briquette be clean and free from grains of sand or dirt, which would prevent a good bearing. The load should be applied at the rate of 600 lbs. per minute. The average of the briquettes of each sample tested should be taken as the test, excluding any results which are manifestly faulty.

Constancy of Volume.

67. Significance. The object is to develop those qualities which tend to destroy the strength and durability of a cement. As it is highly essential to determine such qualities at once, tests of this character are for the most part made in a very short time, and are known, therefore, as accelerated tests. Failure is revealed by cracking, checking, swelling, or disintegration, or all of these phenomena. A cement which remains perfectly sound is said to be of constant volume.

68. Methods. Tests for constancy of volume are divided into two classes: (1) normal tests, or those made in either air or water maintained at about 21° Cent. (70° Fahr.), and (2) accelerated tests, or those made in air, steam, or water at a temperature of 45° Cent. (115° Fahr.) and upward. The test pieces should be allowed to remain 24 hours in moist air before immersion in water or steam, or preservation in air.

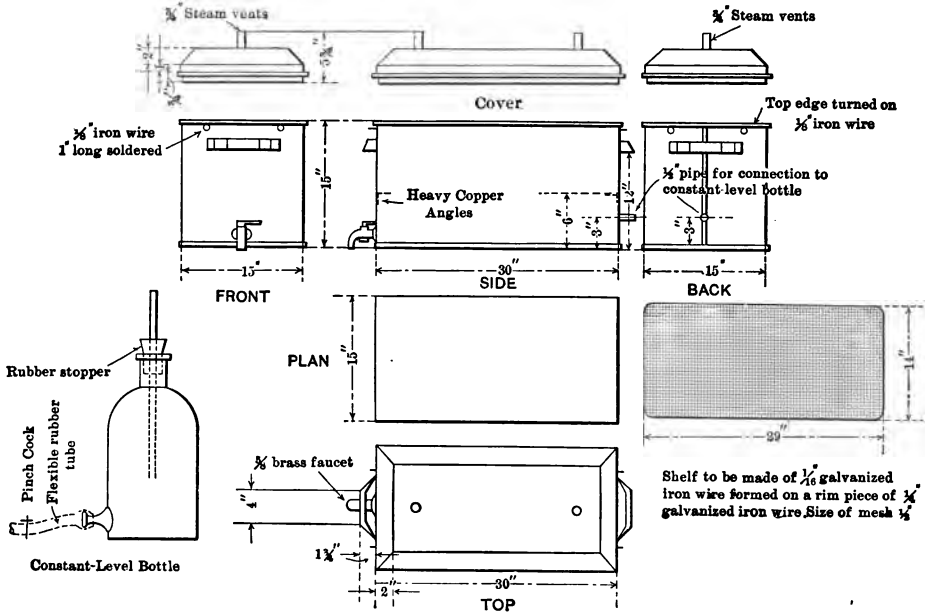

Fig. 28. — Details of Boiler for Accelerated Tests

Boiler is to be made of sheet copper weighing 22 oz. per sq. ft., turned inside. All seams to be lapped where possible. Hard solder to be used only.

69. For these tests, pats, about 7½ cm. (2.95 ins.) in diameter, 1¼ cm. (0.49 in.) thick at the center, and tapering to a thin edge, should be made, upon a clean glass plate [about 10 cm. (3.94 ins.) square], from cement paste of normal consistency.

70. NORMAL TEST. A pat is immersed in water maintained as near 21° Cent. (70° Fahr.) as possible for 28 days, and observed at intervals. A similar pat, after 24 hours in moist air, is maintained in air at ordinary temperature and observed at intervals.

71. ACCELERATED TEST. A pat is exposed in any convenient way in an atmosphere of steam, above boiling water, in a loosely closed vessel, for 5 hours. The apparatus recommended for making these determinations is shown in Fig. 28.

72. To pass these tests satisfactorily, the pats should remain firm and hard, and show no signs of cracking, distortion, or disintegration.

73. Should the pat leave the plate, distortion may be detected best with a straight-edge applied to the surface which was in contact with the plate.

74. In the present state of our knowledge it cannot be said that cement should necessarily be condemned simply for failure to pass the accelerated tests; nor can a cement be considered entirely satisfactory, simply because it has passed these tests.

APPENDIX II.

REPORT OF COMMITTEE ON STANDARD SPECIFICATIONS FOR CEMENT OF THE AMERICAN SOCIETY FOR TESTING MATERIALS. ADOPTED BY THE SOCIETY, NOVEMBER 14, 1904.*

GENERAL OBSERVATIONS.

1. These remarks have been prepared with a view of pointing out the pertinent features of the various requirements and the precautions to be observed in the interpretation of the results of the tests.

2. The Committee would suggest that the acceptance or rejection under these specifications be based on tests made by an experienced person having the proper means for making the tests.

SPECIFIC GRAVITY.

3. Specific gravity is useful in detecting adulteration or under-burning. The results of tests of specific gravity are not necessarily conclusive as an indication of the quality of a cement, but when in combination with the results of other tests may afford valuable indications.

FINENESS.

4. The sieves should be kept thoroughly dry.

TIME OF SETTING.

5. Great care should be exercised to maintain the test pieces under as uniform conditions as possible. A sudden

* Authorized reprint from the copyrighted Proceedings of the American Society for Testing Materials, Volume IV, 1904.

change or wide range of temperature in the room in which the tests are made, a very dry or humid atmosphere, and other irregularities, vitally affect the rate of setting.

Tensile Strength.

6. Each consumer must fix the minimum requirements for tensile strength to suit his own conditions. They shall, however, be within the limits stated.

Constancy of Volume.

7. The tests for constancy of volume are divided into two classes, the first normal, the second accelerated. The latter should be regarded as a precautionary test only, and not infallible. So many conditions enter into the making and interpreting of it that it should be used with extreme care.

8. In making the pats the greatest care should be exercised to avoid initial strains due to molding or to too rapid drying-out during the first twenty-four hours. The pats should be preserved under the most uniform conditions possible, and rapid changes of temperature should be avoided.

9. The failure to meet the requirements of the accelerated tests need not be sufficient cause for rejection. The cement may, however, be held for twenty-eight days, and a retest made at the end of that period. Failure to meet the requirements at this time should be considered sufficient cause for rejection, although in the present state of our knowledge it cannot be said that such failure necessarily indicates unsoundness, nor can the cement be considered entirely satisfactory simply because it passes the tests.

GENERAL CONDITIONS.

10. All cement shall be inspected.

11. Cement may be inspected either at the place of manu facture or on the work.

12. In order to allow ample time for inspecting and

testing, the cement should be stored in a suitable weather-tight building having the floor properly blocked or raised from the ground.

13. The cement shall be stored in such a manner as to permit easy access for proper inspection and identification of each shipment.

14. Every facility shall be provided by the contractor, and a period of at least twelve days allowed for the inspection and necessary tests.

15. Cement shall be delivered in suitable packages with the brand and name of manufacturer plainly marked thereon.

16. A bag of cement shall contain 94 pounds of cement net. Each barrel of Portland cement shall contain 4 bags, and each barrel of natural cement shall contain 3 bags of the above net weight.

17. Cement failing to meet the seven-day requirements may be held awaiting the results of the twenty-eight-day tests before rejection.

18. All tests shall be made in accordance with the methods proposed by the Committee on Uniform Tests of Cement of the American Society of Civil Engineers, presented to the Society January 21, 1903, and amended January 20, 1904, with all subsequent amendments thereto.

19. The acceptance or rejection shall be based on the following requirements:

NATURAL CEMENT.

20. *Definition.* This term shall be applied to the finely pulverized product resulting from the calcination of an argillaceous limestone at a temperature only sufficient to drive off the carbonic acid gas.

Specific Gravity.

21. The specific gravity of the cement thoroughly dried at 100° C. shall be not less than 2.8.

Fineness.

22. It shall leave by weight a residue of not more than 10 per cent on the No. 100, and 30 per cent on the No. 200 sieve.

Time of Setting.

23. It shall develop initial set in not less than ten minutes, and hard set in not less than thirty minutes, nor more than three hours.

Tensile Strength.

24. The minimum requirements for tensile strength for briquettes one inch square in cross-section shall be within the following limits, and shall show no retrogression in strength within the periods specified: *

Age.	*Neat Cement.*	*Strength.*
24 hours	in moist air	50–100 lbs.
7 days	(1 day in moist air, 6 days in water)	100–200 "
28 days	(1 day in moist air, 27 days in water)	200–300 "
	One Part Cement, Three Parts Standard Sand.	
7 days	(1 day in moist air, 6 days in water)	25–75 "
28 days	(1 day in moist air, 27 days in water)	75–150 "

Constancy of Volume.

25. Pats of neat cement about three inches in diameter, one-half inch thick at center, tapering to a thin edge, shall be kept in moist air for a period of twenty-four hours.

(*a*) A pat is then kept in air at normal temperature.

(*b*) Another is kept in water maintained as near 70° F. as practicable.

26. These pats are observed at intervals for at least 28 days, and, to satisfactorily pass the tests, should remain firm and hard and show no signs of distortion, checking, cracking, or disintegrating.

* For example, the minimum requirement for the twenty-four-hour neat cement test should be some specified value within the limits of 50 and 100 pounds, and so on for each period stated.

PORTLAND CEMENT.

27. *Definition.* This term is applied to the finely pulverized product resulting from the calcination to incipient fusion of an intimate mixture of properly proportioned argillaceous and calcareous materials, and to which no addition greater than 3 per cent has been made subsequent to calcination.

Specific Gravity.

28. The specific gravity of the cement, thoroughly dried at 100° C., shall be not less than 3.10.

Fineness.

29. It shall leave by weight a residue of not more than 8 per cent on the No. 100, and not more than 25 per cent on the No. 200 sieve.

Time of Setting.

30. It shall develop initial set in not less than thirty minutes, but must develop hard set in not less than one hour, nor more than ten hours.

Tensile Strength.

31. The minimum requirements for tensile strength for briquettes one inch square in section shall be within the following limits, and shall show no retrogression in strength within the periods specified: *

Age.	*Neat Cement.*	*Strength.*
24 hours in moist air		150–200 lbs.
7 days (1 day in moist air, 6 days in water)		450–550 "
28 days (1 day in moist air, 27 days in water)		550–650 "
	One Part Cement, Three Parts Sand.	
7 days (1 day in moist air, 6 days in water)		150–200 "
28 days (1 day in moist air, 27 days in water)		200–300 "

* For example, the minimum requirement for the twenty-four-hour neat cement test should be some specified value within the limits of 150 and 200 pounds, and so on for each period stated.

Constancy of Volume.

32. Pats of neat cement about three inches in diameter, one-half inch thick at the center, and tapering to a thin edge, shall be kept in moist air for a period of twenty-four hours.

(*a*) A pat is then kept in air at normal temperature and observed at intervals for at least 28 days.

(*b*) Another pat is kept in water maintained as near 70° F. as practicable, and observed at intervals for at least 28 days.

(*c*) A third pat is exposed in any convenient way in an atmosphere of steam, above boiling water, in a loosely closed vessel for five hours.

33. These pats, to satisfactorily pass the requirements, shall remain firm and hard and show no signs of distortion, checking, cracking, or disintegrating.

Sulphuric Acid and Magnesia.

34. The cement shall not contain more than 1.75 per cent of anhydrous sulphuric acid (SO_3), nor more than 4 per cent of magnesia (MgO).

APPENDIX III.

METHOD SUGGESTED FOR THE ANALYSIS OF LIMESTONES, RAW MIXTURES, AND PORTLAND CEMENTS BY THE COMMITTEE ON UNIFORMITY IN TECHNICAL ANALYSIS OF THE NEW YORK SECTION OF THE SOCIETY FOR CHEMICAL INDUSTRY.*

SOLUTION.

One-half gram of the finely powdered substance is to be weighed out and, if a limestone or unburned mixture, strongly ignited in a covered platinum crucible over a strong blast for 15 minutes, or longer if the blast is not powerful enough to effect complete conversion to a cement in this time. It is then transferred to an evaporating dish, preferably of platinum for the sake of celerity in evaporation, moistened with enough water to prevent lumping, and 5 to 10 c.c. of strong HCl added and digested with the aid of gentle heat and agitation until solution is complete. Solution may be aided by light pressure with the flattened end of a glass rod.† The solution is then evaporated to dryness, as far as this may be possible on the bath.

* Reprinted from Standard Methods of Testing and Specifications for Cement, edited by the Secretary, under the direction of Committee C on Standard Specifications of Cement of the American Society for Testing Materials.

† If anything remains undecomposed it should be separated, fused with a little Na_2CO_3, dissolved and added to the original solution. Of course a small amount of separated non-gelatinous silica is not to be mistaken for undecomposed matter.

SILICA (SiO_2).

The residue without further heating is treated at first with 5 to 10 c.c. of strong HCl, which is then diluted to half strength or less, or upon the residue may be poured at once a larger volume of acid of half strength. The dish is then covered and digestion allowed to go on for 10 minutes on the bath, after which the solution is filtered and the separated silica washed thoroughly with water. The filtrate is again evaporated to dryness, the residue without further heating taken up with acid and water, and the small amount of silica it contains separated on another filter paper. The papers containing the residue are transferred wet to a weighed platinum crucible, dried, ignited, first over a Bunsen burner until the carbon of the filter is completely consumed, and finally over the blast for 15 minutes and checked by a further blasting for 10 minutes or to constant weight. The silica, if great accuracy is desired, is treated in the crucible with about 10 c.c. of HFl and four drops of H_2SO_4 and evaporated over a low flame to complete dryness. The small residue is finally blasted, for a minute or two, cooled and weighed. The difference between this weight and the weight previously obtained gives the amount of silica.*

ALUMINA AND IRON (Al_2O_3 AND Fe_2O_3).

The filtrate, about 250 c.c., from the second evaporation for SiO_2, is made alkaline with NH_4OH after adding HCl, if need be, to insure a total of 10 to 15 c.c. strong acid, and boiled to expel excess of NH_3, or until there is but a faint odor of it, and the precipitate iron and aluminum hydrates, after settling, are washed once by decantation and slightly on the filter. Setting aside the filtrate, the precipitate is dissolved in hot dilute HCl, the solution passing into the beaker in which the precipitation was made. The aluminum and

* For ordinary control in the plant laboratory this correction may, perhaps, be neglected; the double evaporation never.

iron are then reprecipitated by NH_4OH, boiled, and the second precipitate collected and washed on the same filter used in the first instance. The filter paper, with the precipitate, is then placed in a weighed platinum crucible, the paper burned off and the precipitate ignited and finally blasted 5 minutes, with care to prevent reduction, cooled and weighed as $Al_2O_3 + Fe_2O_3$.*

IRON (Fe_2O_3).

The combined iron and aluminum oxides are fused in a platinum crucible at a very low temperature with about 3 to 4 grams of $KHSO_4$, or, better, $NaHSO_4$, the melt taken up with so much dilute H_2SO_4 that there shall be no less than 5 grams absolute acid and enough water to effect solution on heating. The solution is then evaporated and eventually heated till acid fumes come off copiously. After cooling and redissolving in water the small amount of silica is filtered out, weighed and corrected by HFl and H_2SO_4.† The filtrate is reduced by zinc, or preferably by hydrogen sulphide, boiling out the excess of the latter afterwards while passing CO, through the flask, and titrated with permanganate.‡ The strength of the permanganate solution should not be greater than .0040 gr. Fe_2O_3 per c.c.

LIME (CaO).

To the combined filtrate from the $Al_2O_3 + Fe_2O_3$ precipitate a few drops of NH_4OH are added, and the solution brought to boiling. To the boiling solution 20 c.c. of a satu-

* This precipitate contains TiO_2, P_2O_5, Mn_3O_4.

† This correction of Al_2O_3 Fe_2O_3 for silica should not be made when the HFl correction of the main silica has been omitted, unless that silica was obtained by only one evaporation and filtration. After two evaporations and filtrations 1 to 2 mg. of SiO are still to be found with the Al_2O_3 Fe_2O_3.

‡ In this way only is the influence of titanium to be avoided and a correct result obtained for iron.

rated solution of ammonium oxalate are added, and the boiling continued until the precipitated CaC_2O_4 assumes a well-defined granular form. It is then allowed to stand for 20 minutes, or until the precipitate has settled, and then filtered and washed. The precipitate and filter are placed wet in a platinum crucible, and the paper burned off over a small flame of a Bunsen burner. It is then ignited, redissolved in HCl, and the solution made up to 100 c.c. with water. Ammonia is added in slight excess, and the liquid is boiled. If a small amount of Al_2O_3 separates, this is filtered out, weighed, and the amount added to that found in the first determination, when greater accuracy is desired. The lime is then reprecipitated by ammonium oxalate, allowed to stand until settled, filtered, and washed,* weighed as oxide by ignition and blasting in a covered crucible to constant weight, or determined with dilute standard permanganate.†

MAGNESIA (MgO).

The combined filtrates from the calcium precipitates are acidified with HCl and concentrated on the steam bath to about 150 c.c., 10 c.c. of saturated solution of $Na(NH_4)HPO_4$ are added, and the solution boiled for several minutes. It is then removed from the flame and cooled by placing the beaker in ice water. After cooling, NH_4OH is added drop by drop with constant stirring until the crystalline ammonium-magnesium ortho-phosphate begins to form, and then in moderate excess, the stirring being continued for several minutes. It is then set aside for several hours in a cool atmosphere and filtered. The precipitate is redissolved in hot dilute HCl, the solution made up to about 100 c.c., 1 c.c. of a saturated solution of $Na(NH_4)HPO_4$ added, and ammonia

* The volume of wash-water should not be too large; vide Hildebrand.

† The accuracy of this method admits of criticism, but its convenience and rapidity demand its insertion.

drop by drop, with constant stirring until the precipitate is again formed as described and the ammonia is in moderate excess. It is then allowed to stand for about 2 hours, when it is filtered on a paper or a Gooch crucible, ignited, cooled, and weighed as $Mg_2P_2O_7$.

ALKALIES (K_2O AND Na_2O).

For the determination of the alkalies, the well-known method of Prof. J. Lawrence Smith is to be followed, either with or without the addition of $CaCO_3$ with NH_4Cl.

ANHYDROUS SULPHURIC ACID (SO_3).

One gram of the substance is dissolved in 15 c.c. of HCl, filtered, and residue washed thoroughly.*

The solution is made up to 250 c.c. in a beaker and boiled. To the boiling solution 10 c.c. of a saturated solution of $BaCl_2$ is added slowly drop by drop from a pipette and the boiling continued until the precipitate is well formed, or digestion on the steam bath may be substituted for the boiling. It is then set aside over night, or for a few hours, filtered, ignited, and weighed as $BaSO_4$.

TOTAL SULPHUR.

One gram of the material is weighed out in a large platinum crucible and fused with Na_2CO_3 and a little KNO_3, being careful to avoid contamination from sulphur in the gases from source of heat. This may be done by fitting the crucible in a hole in an asbestos board. The melt is treated in the crucible with boiling water, and the liquid poured into a tall narrow beaker and more hot water added until the mass is disintegrated. The solution is then filtered. The

* Evaporation to dryness is unnecessary, unless gelatinous silica should have separated and should never be performed on a bath heated by gas; vide Hildebrand.

filtrate contained in a No. 4 beaker is to be acidulated with HCl and made up to 250 c.c. with distilled water, boiled, the sulphur precipitated as $BaSO_4$ and allowed to stand over night or for a few hours.

LOSS ON IGNITION.

Half a gram of cement is to be weighed out in a platinum crucible, placed in a hole in an asbestos board so that about $\frac{3}{8}$ of the crucible projects below, and blasted 15 minutes, preferably with an inclined flame. The loss by weight, which is checked by a second blasting of 5 minutes, is the loss on ignition.

May, 1903: Recent investigations have shown that large errors in results are often due to the use of impure distilled water and reagents. The analyst should, therefore, test his distilled water by evaporation and his reagents by appropriate tests before proceeding with his work.

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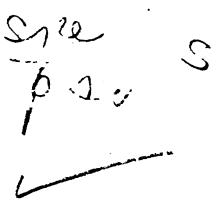

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